



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification <sup>6</sup> : G01N 3/46, G01J 3/44, G01B 11/00</p>	<p>A1</p>	<p>(11) International Publication Number: <b>WO 97/03346</b> (43) International Publication Date: 30 January 1997 (30.01.97)</p>
<p>(21) International Application Number: PCT/GB96/01658 (22) International Filing Date: 12 July 1996 (12.07.96) (30) Priority Data: 9514367.3 13 July 1995 (13.07.95) GB (71) Applicant (for all designated States except US): RENISHAW PLC [GB/GB]; New Mills, Wotton-under-Edge, Gloucestershire GL12 8JR (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): PITT, Gillies, David [GB/GB]; Glen Sunar, Brockweir Road, Hewelsfield, Gloucestershire GL15 6UU (GB). HAYWARD, Ian, Paul [GB/GB]; 6 Deanhead Grove, Clifton, York, Yorkshire YO3 4UH (GB). (74) Agent: JACKSON, John, Timothy; Renishaw plc, Patent Dept., New Mills, Wotton-under-Edge, Gloucestershire GL12 8JR (GB).</p>		<p>(81) Designated States: JP, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  Published With international search report.</p>
<p>(54) Title: STRAIN AND/OR STRESS SENSITIVE DEVICES</p> <div data-bbox="396 1163 1185 1797"> </div> <p>(57) Abstract</p> <p>A probe for scratch, indentation or tribological testing, or a contact-detecting probe for dimensional measurement, comprises a stylus (14) with a tip (74). Illuminating light delivered by an optical fibre (70) causes Raman scattering by the material of the tip (74), which passes back through the optical fibre (70) for analysis. When the tip (74) contacts a sample (10), it suffers stress and/or strain. This stress and/or strain causes changes in the Raman scattered light, which are detected.</p>		

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MW	Malawi
AT	Austria	GE	Georgia	MX	Mexico
AU	Australia	GN	Guinea	NE	Niger
BB	Barbados	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IE	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KE	Kenya	RO	Romania
BY	Belarus	KG	Kyrgyzstan	RU	Russian Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	KZ	Kazakhstan	SG	Singapore
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SZ	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Latvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

## STRAIN AND/OR STRESS SENSITIVE DEVICES

This invention relates to devices which are sensitive to strain and/or stress.

5

It is known to provide a probe of the type used on coordinate measuring machines or machine tools with a strain sensor. These may be arranged to undergo strain, and thus provide an output, when a stylus tip of the probe comes into contact with a workpiece. Examples are shown in European Patent Applications EP 0068899 and EP 0243766.

It is also known that strain or stress in a sample can be detected by means of various effects which the strain or stress has upon light which is Raman scattered when the sample is illuminated, e.g. by a laser. In co-pending International Patent Application No. WO96/10737, which has a common priority date with the present application, use is made of this effect to detect stresses and/or strains induced in a sample after it has been subjected to an indentation or scratch test.

The present invention provides a probe having a tip for contacting a workpiece; means for illuminating the tip so as to produce Raman scattered light from the tip; and means for detecting the Raman scattered light and determining therefrom the stress or strain in the tip.

Such a probe has a wide variety of uses, including scratch or indentation testing; tribological testing; and touch probes of the type used on coordinate measuring machines or machine tools.

Preferred embodiments of the present invention will now be described by way of example, with reference to the accompanying drawings, wherein:

Fig 1 is a schematic diagram of Raman detection apparatus;

Fig 2 is a partly sectional view of a probe for use in scratch or indentation testing;

Fig 3 is a plan view of apparatus for tribological testing;

5 Fig 4 is a partly sectional view of a touch probe for use in a coordinate measuring machine or machine tool;

Figs 5 and 6 are schematic diagrams showing part of alternative arrangements of the Raman detection apparatus of Fig 1; and

10 Fig 7 shows a modification of Fig 2.

Fig 1 shows a Raman spectrometer, much of which is as described in European Patent Application No. EP 0543578 (to which the reader should refer for more detail). Light from  
15 a laser source 30 is directed via mirrors 32,34 and a holographic notch filter 38. This directs the laser light down an optical fibre 70 towards a sample. Raman scattered light passes back through the optical fibre 70 and is transmitted by the holographic notch filter 38, which  
20 rejects reflected and Rayleigh scattered light having the same frequency as the laser source 30. A further such holographic notch filter 44 may be provided to improve the rejection of the Rayleigh light if required. A rugate filter or filters may be used in place of the filters  
25 38,44. The scattered Raman spectrum is then analysed by an analyser 46, and is focused by a lens 48 on to a detector such a cooled charged coupled device (CCD) 50. The analyser 46 is suitably a diffraction grating, which disperses the spectrum across the CCD 50. However, it is  
30 also possible to use a tunable filter, which selects light of a particular Raman wavenumber and passes it to the CCD 50. The data from the CCD is acquired by a computer 54 for further analysis. This computer may also control the Raman analyser 46.

35

Fig 2 shows one possible arrangement at the other end of the optical fibre 70 of Fig 1. The equipment shown in Fig 2 is basically a scratch or indentation or nano-indentation

tester, for example of the type described by H.E.

Hintermann, in "Characterisation of surface coatings by the scratch adhesion test and by indentation measurements",

Fresenius Journal of Analytical Chemistry (1993), 346:45-

5 52. Such machines are available commercially from Centre Suisse d'Electronique et de Microtechnique SA (CSEM), Rue de la Maladière 71, CH 2007, Neuchâtel, Switzerland.

As shown schematically in Fig 2, an indentation is made  
10 into the surface of a sample 10 placed on a table 12, by means of a diamond tip 74 at the end of a stylus 14. The load L which produces the scratch or indentation is variable with time, and is applied by a motor (not shown) via a lever 28 having a fulcrum 29. Thus, in the case of a  
15 scratch tester, the sample 10 is moved horizontally under the tip 74 by means of the table 12, and a gradually increasing force is applied to scratch the surface. The tip may have any desired shape, e.g. conical, hemi-spherical or pyramid-shaped. Instead of diamond, it may be  
20 made of another suitable material such as  $Al_2O_3$  or sapphire. The scratch or indentation test is especially useful when the sample 10 has a very thin coating or surface film (not shown), as explained in the above paper by Hintermann.

25 The commercially available scratch or indentation tester is modified in that the stylus 14 is tubular, having the optical fibre 70 passing down it. The tip 74 has a flat, polished upper surface 75. The laser light from the laser source 30 is delivered by the optical fibre 70 and focused  
30 by a lens 72 through the surface 75 onto the tip 74.

Preferably it is focused to the point 76 which is the interface between the tip 74 and the sample 10. Raman scattered light from this same point is then collected by the lens 72 and passes back along the optical fibre 70 and  
35 through the holographic filter 38, to be analysed by the Raman analyser 46 and detected by the CCD 50.

Corresponding data is acquired by the computer 54.

In use, Raman spectra are acquired by the computer 54 at numerous sample times during the course of a normal scratch or indentation test on a sample 10. It is preferable that the scratch or indentation tests should be controlled by the same computer 54 which controls the Raman detection, to facilitate the correlation of the times at which the various Raman spectra are acquired with the corresponding times and/or positions on the sample 10 of data normally provided during the scratch or indentation test. For example, it is desirable to correlate the different Raman spectra with the instantaneous applied load L. The Raman data may be analysed to determine instantaneous values of strain or stress present either in the sample 10, or in the diamond tip 74, or both. To do this, use is made of a preselected Raman peak or peaks characteristic of the material of the sample 10 or of the diamond of the tip 74. In particular, strain/stress dependent properties of such a peak or peaks are monitored, such as an increase in the width of the peak or a shift of the peak to another wavenumber when strain is present. Of course, the tip 74 may be made from another material rather than diamond, with a suitable strain/stress-sensitive Raman peak which is monitored.

The computer 54 is then able to output information, e.g. in graphical form, showing how the strains and stresses induced in the sample 10 vary during the cycle of an indentation or scratch test. As discussed in co-pending International Patent Application No. WO96/10737, changes in this data indicate critical points in the behaviour of the sample. For example, where the sample comprises a coating or film upon a substrate, the stress/strain data derived may indicate critical points at which there is a breakdown in the cohesion or adhesion of the film or coating. Effects such as plastic deformation of the material of the film or coating may also be determined.

Advantageously, the Raman spectrometer in Fig 1 may make use of confocal techniques. This may involve the use of a pinhole as described by G J Puppels et al, Nature, Vol. 347, (20 Sept 1990) pp 301-303, or may be as described in International Patent Application No. WO 92/22793. The Raman scattered light which is analysed and detected may thereby be restricted to a small depth of field, say about 10 $\mu$ m in the region of the point 76. This ensures that the spectrometer is sensitive only to the stressed or strained material of the tip 74 and/or of the sample 10 within this small region.

Where the sample 10 comprises a thin film or coating on a substrate, it is possible to determine not only stress/strain data for the tip 74 and for the material of the coating, but also for the underlying substrate material, by monitoring suitable Raman peaks characteristic of the coating and the substrate respectively. This yields still further information about the properties of the interface between the coating and the substrate.

If desired, it is possible to determine strain only of the tip 74, or only of the sample 10, by restricting the analysis to corresponding peaks in the Raman spectrum. In these cases, the lens 72 may be focused at a point within the tip 74, or within the sample 10, rather than at the interface point 76.

The stress/strain data for the material of the tip 74 is potentially useful because it gives a very direct indication of the instantaneous load applied at the point 76 to the sample 10.

Fig 3 is a plan view of apparatus for tribological testing of a sample 80 which is in the form of a disc. The sample 80 is mounted on a turntable 82 and rotated in the direction indicated by an arrow 84. A pin 86 is mounted vertically at the end of a horizontal lever arm 88, and has

a tip which presses down on the surface of the sample 80 as it rotates, producing a wear track 90. The lever arm 88 is mounted about a pivot 92, and the drag between the pin 86 and the sample 80 may be determined by measuring lateral forces on the lever 88 e.g. as indicated by arrows 94.

The tip at the lower end of the pin 86 may be of diamond or another suitable material, shaped in any conventional manner as commonly used in such pin-on-disc tribological testing. However the pin 86 is constructed in a similar manner to the stylus 14 in Fig 2. Thus, it is tubular and has the optical fibre 70 passing down it. Light from the laser source 30 (Fig 1) is directed to the tip of the pin 86 down the optical fibre 70, and Raman scattered light passes back up the optical fibre to the Raman analyser 46 and CCD detector 50, as previously. It is also possible to use confocal techniques as described previously.

Analysis of the resulting Raman data yields information about the strains and stresses experienced either in the tip of the pin 86, or in the material of the sample 80, or both. It may also give data on the composition of wear debris in the track 90, or on the breakdown of any lubricant which may be used, by monitoring appropriate parts of the Raman spectrum.

Raman data may also be obtained in the same way from a pin similar to the pin 86, but mounted in tribological apparatus of the type in which the pin undergoes reciprocating motion relative to the sample, rather than rotary motion.

Fig 4 shows a dimensional measurement probe of the type used on coordinate measuring machines and machine tools. Such probes are known, for example, from U.S. Patent No. 4,153,998. They comprise a housing 100, from which extends a deflectable stylus 102 having a tip 104. This can be brought into contact with a workpiece 106 by movement of



the probe 100, e.g. as suggested by the arrow 108. It may also be brought into contact with the workpiece by a horizontal movement, e.g. in order to contact a vertical surface of the workpiece. The stylus 102 may be mounted  
5 within the housing 100 in any conventional manner, such as a kinematic mount 110 of the type described in U.S. Patent No 4,153,998.

To detect the instant of contact with the workpiece 106,  
10 the optical fibre 70 passes down the centre of the stylus 102 and into the material of the stylus tip 104 (which may be a material such as diamond, or may be synthetic ruby as is more conventional in such probes). A lens may be incorporated in the stylus as before, if desired. Raman  
15 scattered light from the material of the tip 104 passes back up through the optical fibre 70, and may be analysed, e.g. using the apparatus of Fig 1. At the instant of contact between the tip 104 and the workpiece 106, strain is induced in the tip 104 and this is detected from any of  
20 the strain/stress related Raman effects mentioned above, and used to generate the required trigger signal.

The probe of Fig 4 may also be used to analyse the composition and/or stress/strain in the workpiece 106, if  
25 desired.

Rather than use the complicated and expensive equipment as shown in Fig 1, it may be modified as shown in Figs 5 and 6. Here, the Raman analyser 46 is replaced by an analyser  
30 46A in the form of a filter, e.g. a holographic filter or dielectric filter. This is pre-tuned to a fixed Raman wavenumber, as discussed below. Light of this wavenumber is focused by the lens 48 onto a detector 50A, which may for example be an avalanche photodiode. In place of the  
35 computer 54, much simpler signal processing circuits 120 may now be used. These simply amplify the signal from the detector 50A and issue a trigger signal when it passes through a predetermined threshold value.

If the filter 46A is a passband filter pre-tuned to a Raman peak of the material of the stylus tip 104 which shifts to a different wavenumber when strain is present, then contact with the workpiece 106 will result in a decrease in the signal received by the detector 50A (caused by the fact that the shifted Raman peak is no longer transmitted by the filter 46A to the same extent). The width of the passband of the filter should preferably be of a similar order to the width of the Raman peak, with a sharp cut-off edge in the direction of the shift. Alternatively, an edge filter with a sharp cut-off pre-tuned to coincide with the unshifted Raman peak may be used (see European Patent Application No. 96303849.2, US Patent Application No. 08/656,691 and Japanese Patent Application No. 146,330/1996, especially the description relating to Fig 5 of those applications).

Alternatively, if the filter 46A is pre-tuned to the shifted Raman wavenumber, so that it passes a relatively low intensity at the unshifted wavenumber, then the signal processing circuit 120 will register an increase in signal when the stylus 104 contacts the workpiece 106.

Another alternative is to choose a Raman peak which undergoes broadening when the material of the stylus tip 104 is strained. The filter 46A may now be a passband or edge filter with a sharp edge pre-tuned to a wavenumber just above or below the maximum of the chosen Raman peak. When the stylus contacts the workpiece, and the peak is broadened, the intensity of light reaching the detector 50A increases and this may be detected by the circuit 120.

Fig 6 shows an alternative. Here, the analyser is a filter 46B. This may be an edge filter which transmits light above a certain wavenumber via a lens 48A to a detector 50A, while reflecting all light below that wavenumber via a lens 48B to a detector 50B (or vice versa). The edge of this filter may be pre-tuned to a

wavenumber which coincides with or is just slightly to one side of the maximum of the Raman peak of interest. Thus, when the peak shifts (or broadens, as the case might be) there is a substantial differential change between the intensities of light received at the two detectors 50A,50B. This may be detected by a differentially acting signal processing circuit 122, which issues the desired trigger signal. To prevent the effect of light from other parts of the Raman spectrum, a narrow pass-band filter 44A may be provided to reject all light except in a pass-band around the Raman peak of interest.

Fig 7 shows a modified probe, generally similar to that of Fig 2. However, in place of a single optical fibre 70, there are two optical fibres 70A,70B. In practice, a single optical fibre cable having multiple cores may be used.

The optical fibre 70A carries the exciting laser light from a suitable source, without the need for it to be injected into the optical path by the filter 38 (Figs 1,5 or 6). However, within the stylus 14, a filter 38A is provided on the end of the fibre 70A. This is a narrow passband filter which passes only the light of the laser frequency, rejecting all other frequencies. In particular, it rejects Raman-shifted light and fluorescence generated within the fibre 70A itself as a result of interaction between the laser light and the material of the fibre. Such unwanted Raman and fluorescence signals could otherwise cause spurious signals which mask the Raman effects which occur in the stylus tip 74 and sample 10.

As previously, the Raman scattered light from the tip 74 and sample 10 is collected by the lens 72. It is taken to a Raman analysis section according to Figs 1,5 or 6 via the optical fibre 70B. The fibre 70B also has a filter 38B at its end, within the stylus 14. The filter 38B is a narrow notch filter, designed to reject light having the same

frequency as the laser. It therefore rejects Rayleigh scattered and reflected laser light, while passing the desired Raman signals.

- 5 The filter 38B provides further rejection of the strong signal of the laser frequency, assisting the filter 38 (Figs 1,5 and 6) in this respect. Furthermore, it minimises any tendency for spurious Raman and fluorescence signals to be generated within the fibre 70B by interaction  
10 between the laser frequency signal and the material of the fibre 70B.

For further details of optical arrangements in optical fibre probes for Raman purposes, which can be used with the  
15 present invention, reference is made to U.S. Patents 5,112,127 (Carrabba) and 5,377,004 (Owen et al). Both these patents describe optical arrangements which may be incorporated within the stylus 14, in place of the filters 38A,38B and the lens 72. Alternatively, the probes of  
20 these U.S. patents may be mounted adjacent the probe seen in Fig 2, the objective lenses of the probes shown in the US patents being linked to the probe of Fig 2 by a short length of an optical fibre. By keeping this linking fibre short, any tendency to generate spurious Raman or  
25 fluorescence signals within the fibre will be minimised.

In place of the above arrangements, a single filter may be placed on the end of the fibre 70 within the stylus 14 in Fig 2. This should be a notch filter, tuned to the  
30 specific Raman line created by interaction between the exciting laser light and the material of the fibre. If there is more than one such Raman line, then the filter may have a corresponding number of notches at the appropriate wavenumbers to remove them. This can be achieved by having  
35 two or more filters in series, or by providing a single filter tailored to have the appropriate notches. In any case, the function of such a filter is to pass both the exciting laser light from the fibre 70 to the tip 74 and

sample 10, and to pass the required Raman signals from the sample 10 and tip 74 back to the fibre 70.

5 The probe shown in Fig 4 may be modified in any of the above ways to incorporate one or more filters at the interface between the fibre 70 and the stylus tip 104, including the possibility of having two fibres 70A, 70B, and the possibility of using arrangements according to U.S. Patents 5,112,127 or 5,377,004.

## CLAIMS

1. A probe having a tip for contacting a workpiece; means  
for illuminating the tip so as to produce Raman scattered  
5 light from the tip; and means for detecting the Raman  
scattered light and determining therefrom the stress or  
strain in the tip.
2. A probe according to claim 1, including a stylus, said  
10 tip being carried by the stylus, light from said  
illuminating means passing through the stylus to said tip.
3. A probe according to claim 1 or claim 2, including a  
stylus, said tip being carried by the stylus, said Raman  
15 scattered light passing from said tip through the stylus.
4. A probe according to any one of the preceding claims,  
including at least one optical fibre for carrying the  
illuminating light and/or the scattered light to/from said  
20 tip.
5. A probe according to claim 4, having a first optical  
fibre for carrying the illuminating light to the tip, and a  
second optical fibre for carrying the scattered light from  
25 the tip.
6. A probe according to claim 4 or claim 5, including at  
least one filter between the tip and a said optical fibre.
- 30 7. A probe according to any one of the preceding claims,  
being a probe for scratch, indentation or tribological  
testing.
8. A probe according to any one of claims 1 to 6, being a  
35 dimensional measurement probe for detecting contact with a  
workpiece.

9. A probe according to any one of the preceding claims, wherein the detecting and determining means comprises a filter which is tuned to a Raman wavenumber which has a predetermined relation to a Raman peak of the material of
- 5 said tip, said peak being affected by stress or strain in the tip.

1/4

FIG. 1

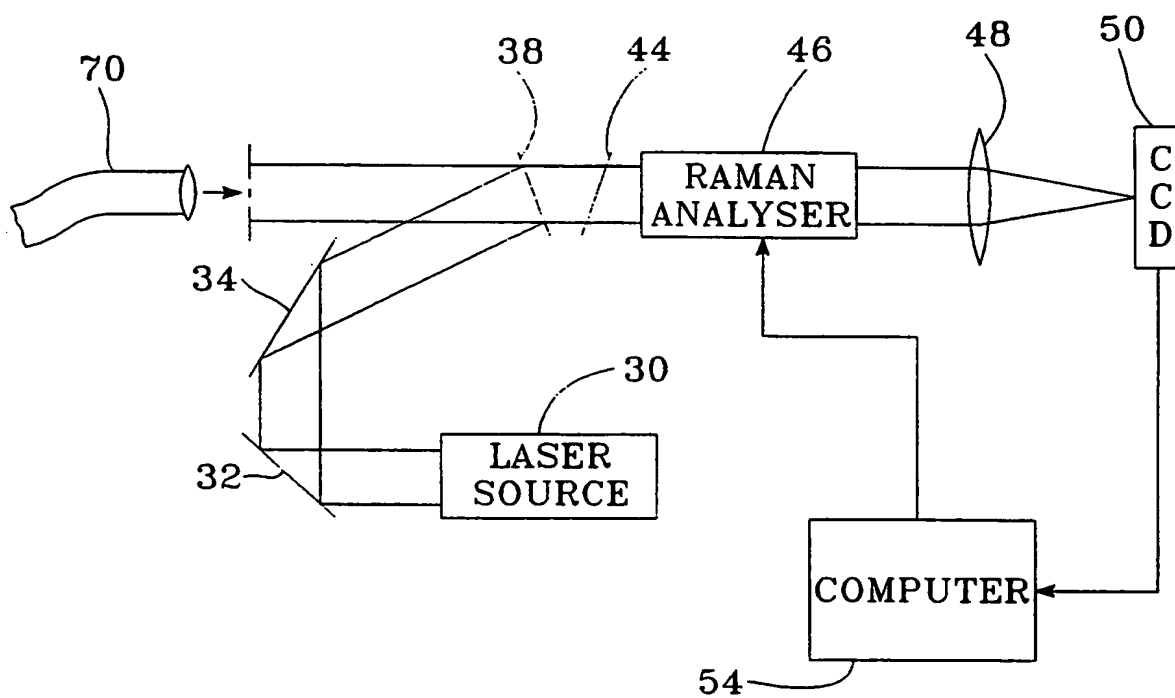
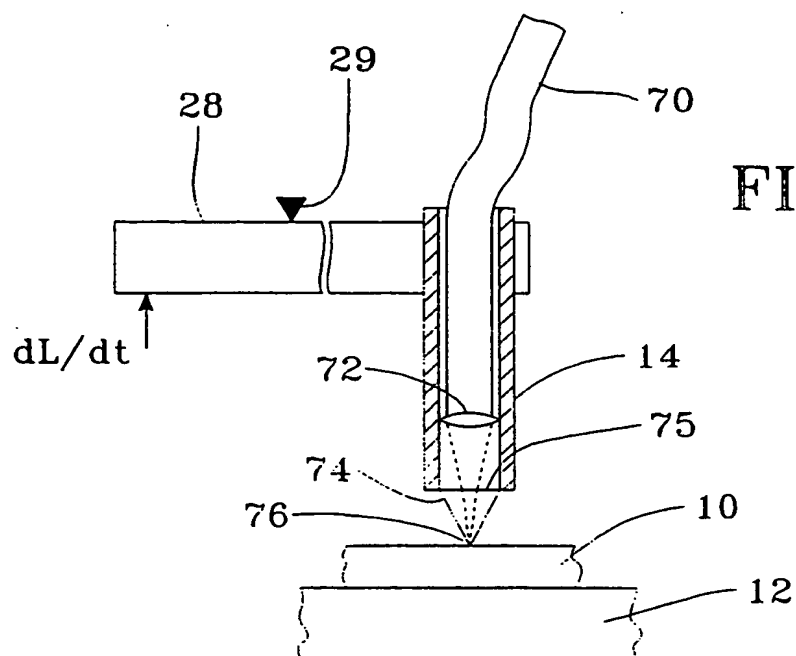


FIG. 2





2/4

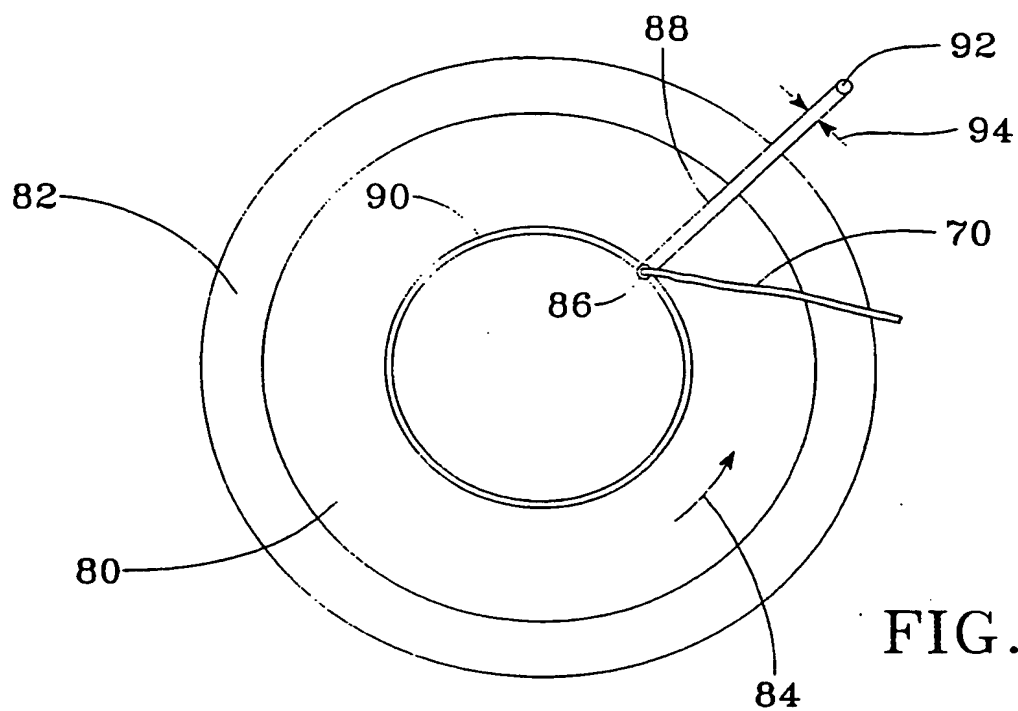


FIG. 3

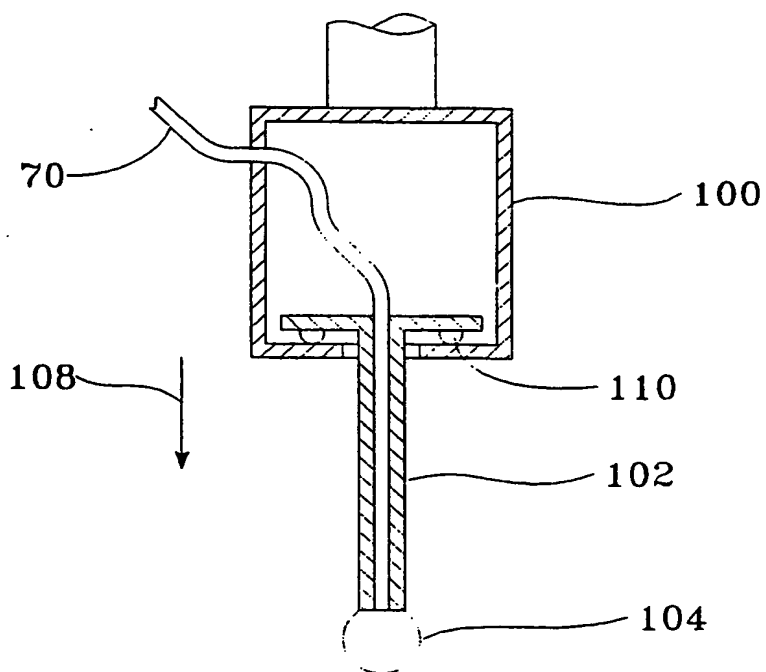
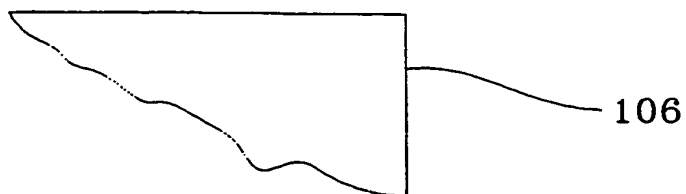


FIG. 4



3/4

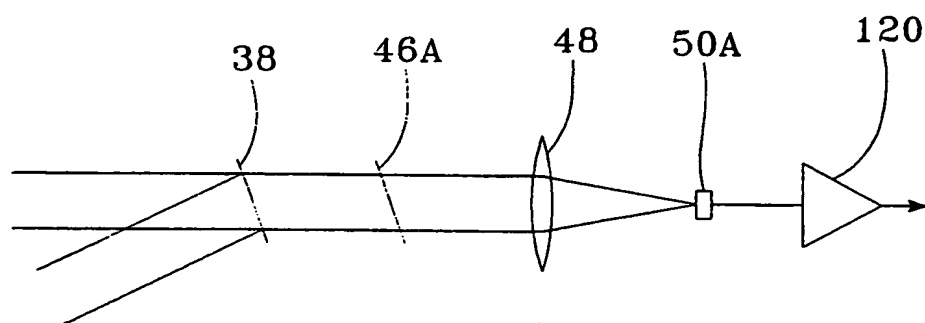


FIG. 5

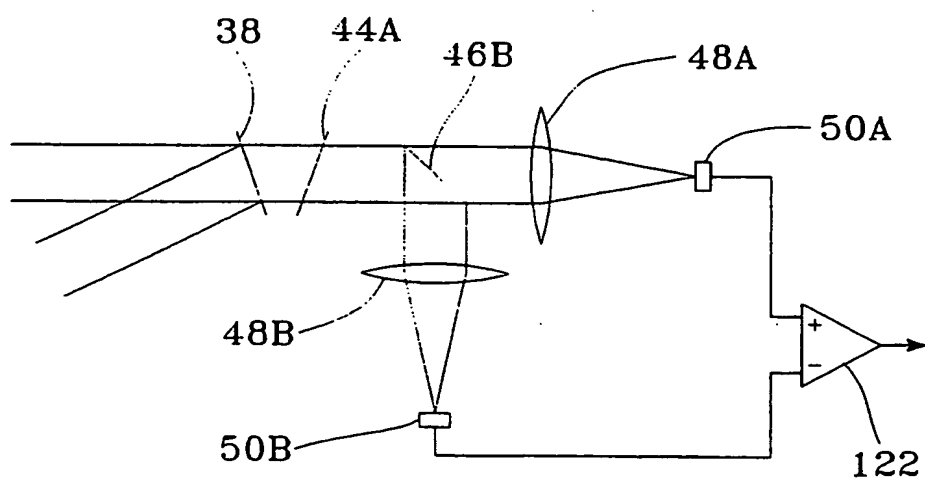


FIG. 6

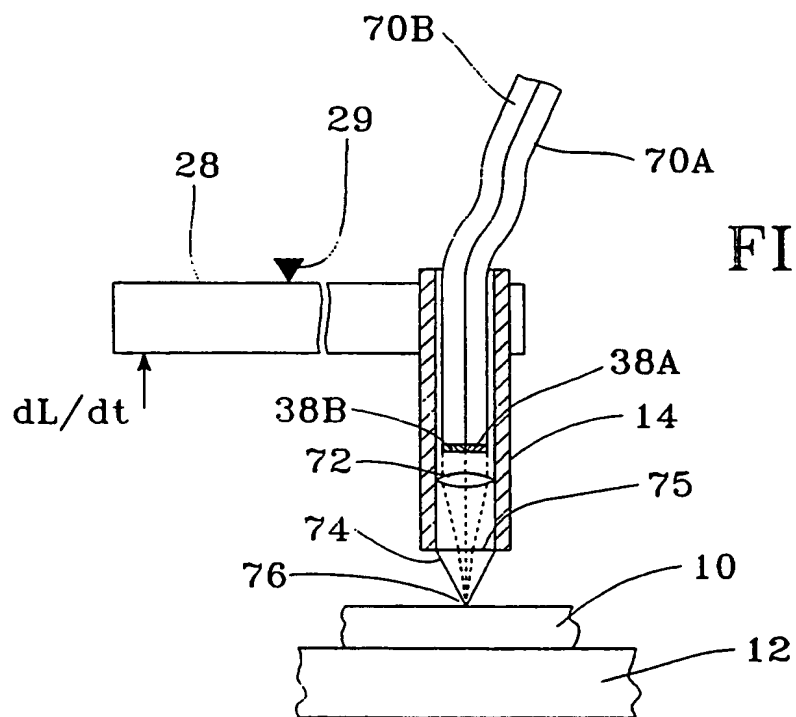


FIG. 7

# INTERNATIONAL SEARCH REPORT

Inter. Appl. No.  
PCT/GB 96/01658

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 G01N3/46 G01J3/44 G01B11/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 G01N G01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO,A,92 22805 (RENISHAW) 23 December 1992 see abstract see page 4, line 10 - line 32 see page 6, line 4 - line 33 see page 11, line 15 - line 17 see figures 1-3	1-7,9
A	--- PATENT ABSTRACTS OF JAPAN vol. 13, no. 478 (P-951), 30 October 1989 & JP,A,01 189544 (SHIMADZU), 28 July 1989, see abstract	1
A	--- EP,A,0 068 899 (ROLLS-ROYCE) 5 January 1983 cited in the application see abstract --- -/--	8

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

5 November 1996

Date of mailing of the international search report

13. 11. 96

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+ 31-70) 340-3016

Authorized officer

Thomas, R.M.

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/GB 96/01658

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	<p>WO,A,96 10737 (RENISHAW) 11 April 1996  cited in the application  see abstract  see page 12, line 4 - line 6  see page 13, line 11 - line 20  see page 15, line 35 - line 37  see page 16, line 6 - line 8  see page 16, line 21 - line 22  see figures 10,12  -----</p>	1-7,9

# INTERNATIONAL SEARCH REPORT

information on patent family members

Int. Application No

PCT/GB 96/01658

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO-A-9222805	23-12-92	DE-D- 69203830	07-09-95
		DE-T- 69203830	23-11-95
		EP-A- 0542962	26-05-93
		WO-A- 9222793	23-12-92
		JP-T- 6500637	20-01-94
		US-A- 5510894	23-04-96
EP-A-0068899	05-01-83	DE-A- 3279249	05-01-89
		JP-B- 1039522	22-08-89
		JP-C- 1556462	23-04-90
		JP-A- 58044302	15-03-83
		US-A- 4462162	31-07-84
WO-A-9610737	11-04-96	EP-A- 0731908	18-09-96